Epoxies for vacuum impregnation of superconducting magnets: A review and assessment of critical properties

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Oct 10, 2018

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U.S. DEPARTMENT OF ENERGY

U.S. MAGNET DEVELOPMENT PROGRAM

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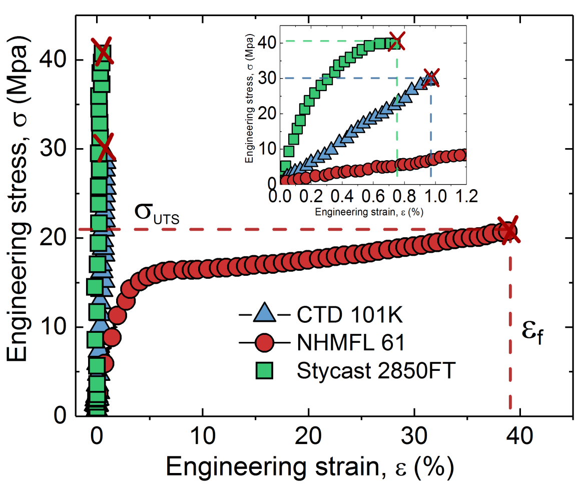
What we discussed last time - CTD101K versus NHMFL 61

Thermal shock test
After one thermal cycle (RT -> 77 K->RT)

CTD101K

NHMFL 61

Room Temperature, test standard - ASTM-D638



NHMFL 61 mix

Properties	CTD-101k	NHMFL-mix61
Chemistry	liquid epoxy resin + anhydride hardener	bisphenol-A based liquid epoxy resin + amine hardener + high molecular weight additive
Elongation at break (%) [RT]	0.97	10.2
Glass transition temperature T_g (° C)	113	65
Radiation hardness	Ok at 30 MGy for high-lumi IR magnets	Unknown
Pros:	Low viscosity, Long pot life	High toughness High thermal shock resistance
Cons:	Brittle Low toughness	Higher viscosity Shorter pot life

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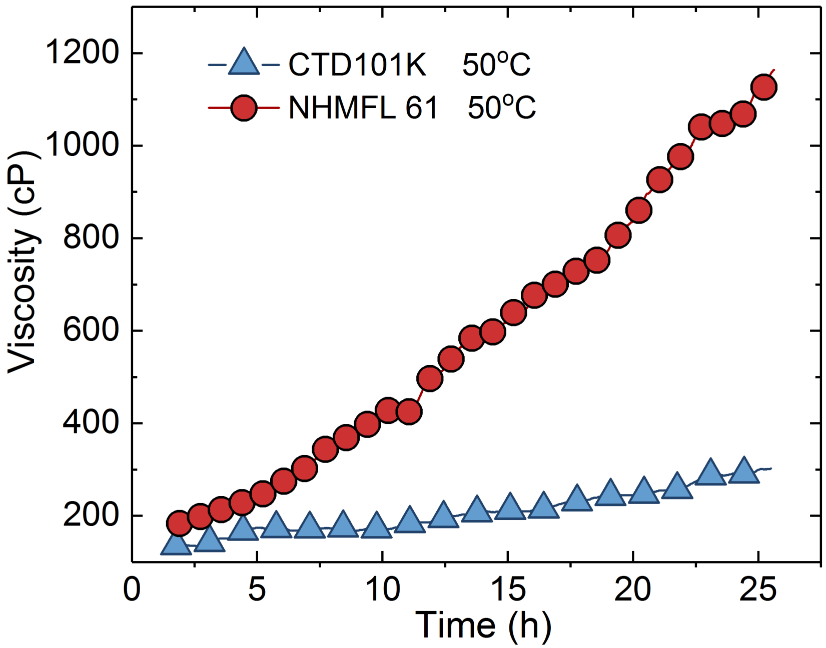
Viscosity test at 50°C



Viscosity test

Review of the resin systems used in major superconducting magnets

Magnet system	*Magnet type and applications*	*Resin*	*Recipe details*	*Notes on potting requirements*	Notes on Resin	*References*
ITER Central Solenoid	Solenoids for Fusion	Epoxy	GY282 + HY918 + DY073	Long pot life required	DGEBF resin + a low reacting anhydride MTHPA hardner	[1,2]
ITER TF coils	D-shaped Magnets for Fusion	Epoxy/Cyanate Ester	CTD425	Long pot life and radiation resistance required	Used with a primer, CTD-450, to ensure adhension. Also used for the NSTX-U central stack.	[3]
ATLAS End Cap Toroid Magnet	Detector Magnets	Epoxy	GY282 + HY5200 + DER732	Long pot life		[4]
NHMFL 900 MHz NMR solenoid	Solenoids	Epoxy	NHMFL mix 61, confidential	High toughness	DGEBA resin + An aromatic amine + A high molecular weight additive	[5]
High-luminosity LHC Nb_3Sn QXF Quadrupole Magnets (LARP/AUP + CERN)	Accelerator magnets	Epoxy	CTD101k	High radiation resistance (30 MGy)	DGEBA resin + an anhydrige hardner	[6]
University of Twente, MSUT Nb_3Sn dipole	Accelerator magnets	Epoxy	MY740 + HY906 + DY062	Reasonable pot life and viscosity	DGEBA resin + an anhydride MTHPA hardner; similar to ITER CS epoxy and CTD101K	[7]
N/A	N/A	Epoxy	Epoxy + Jeffamine amines curing agents	N/A	High toughness	[8,9]

[1] Reed, R., D. Evans, and P. Fabian, Development of a new resin system for the US ITER central solenoid model coil, in Advances in Cryogenic Engineering Materials. 2000, Springer. p. 227-234.
[2] Madhukar, Madhu S., and Nicolai N. Martovetsky. "DGEBF epoxy blends for use in the resin impregnation of extremely large composite parts." Journal of Composite Materials 49.30 (2015): 3741-3753.
[3] Munshi, Naseem A., et al. "Radiation resistant electrical insulation qualified for ITER TF coils." IEEE transactions on applied superconductivity 23.3 (2013): 7700104-7700104.
[4] Reed, R.P., Low-Viscosity, Radiation-Resistant Resin System with Increased Toughness. 2004. 711: p. 209-216.
[5] Markiewicz, W.D., et al., 25 T high resolution NMR magnet program and technology. IEEE Transactions on Magnetics, 1996. 32(4): p. 2586-2589.
[6] Savary, F., et al. "The 11 T dipole for HL-LHC: Status and plan." IEEE Transactions on Applied Superconductivity 26.4 (2016): 1-5.
[7] Den Ouden, A., et al. "An experimental 11.5 T Nb/sub 3/Sn LHC type of dipole magnet." IEEE Transactions on magnetics 30.4 (1994): 2320-2323.
[8] Baldan, Carlos A., and Carlos Y. Shigue. "Development of a new epoxy resin for superconducting magnet impregnation." IEEE transactions on applied superconductivity 10.1 (2000): 1347-1349.
[9] Baldan, C. A., et al. "Study of bisphenol-F epoxy resin system for impregnation of superconducting magnets." Advances in Cryogenic Engineering Materials. Springer, Boston, MA, 2000. 205-210.

Review of the resin systems used in major superconducting magnets – there are actually only four systems.

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[6] Savary, F., et al. "The 11 T dipole for HL-LHC: Status and plan." IEEE Transactions on Applied Superconductivity 26.4 (2016): 1-5.
[7] Den Ouden, A., et al. "An experimental 11.5 T Nb/sub 3/Sn LHC type of dipole magnet." IEEE Transactions on magnetics 30.4 (1994): 2320-2323.
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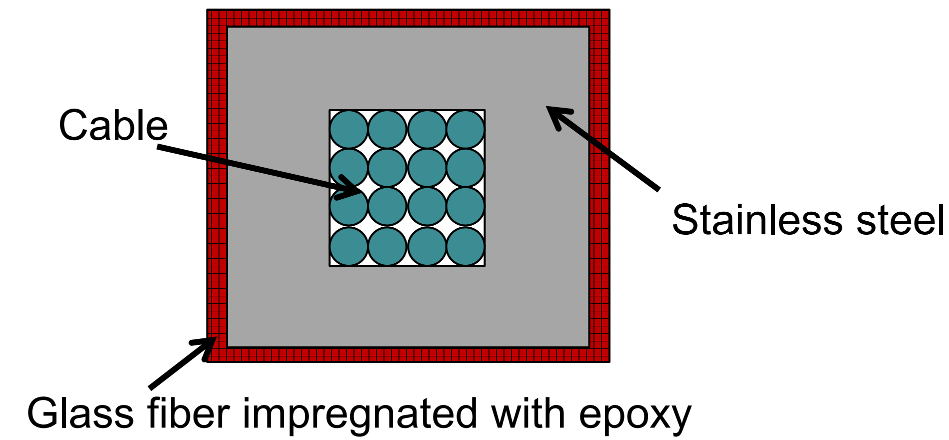
[9] Baldan, C. A., et al. "Study of bisphenol-F epoxy resin system for impregnation of superconducting magnets." Advances in Cryogenic Engineering Materials. Springer, Boston, MA, 2000. 205-210.

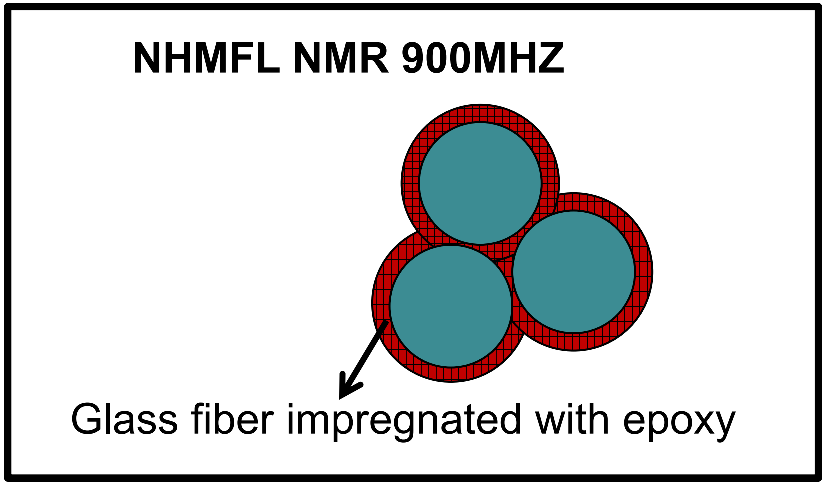
A reminder – Interaction between epoxy and superconducting wires is not always the same

Glass fiber ▦ Epoxy ■

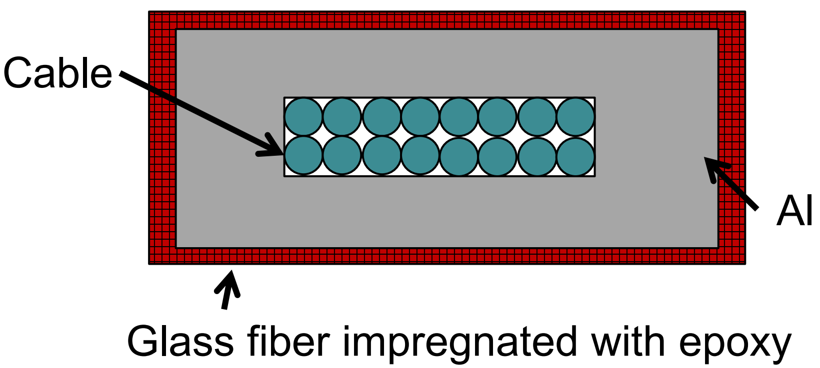
Mostly tensile

ITER Central Solenoid

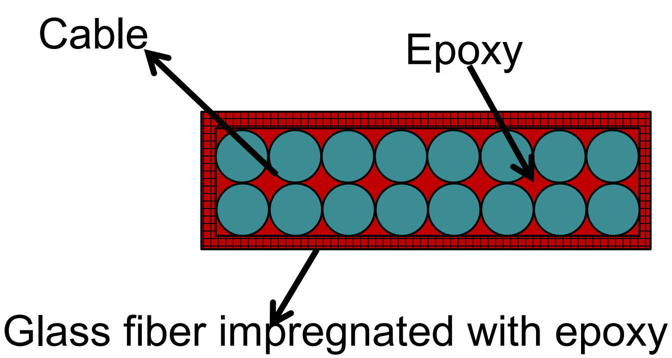




ATLAS End Cap Toroid



Nb_3Sn accelerator magnets



Sample list of tested epoxies – three systems covered.

Color coding the same with that on the slide 5.

Recipe No.	Epoxy	Curing agent	Modifier	Cure
Rcp1	GY282	Jeffamine D230	N/A	80°C@2h 125°C@3h
Rcp2	GY282	Jeffamine D400	N/A	80°C@2h 125°C@3h
Rcp3	GY282	Jeffamine D400/D2000	N/A	80°C@2h 125°C@3h
Rcp4 ATLAS ECT [1]	GY282	HY5200	DER 732 (10 pbw)	130°C@15h
Rcp5 Modified ATLAS ECT [1]	GY282	HY5200	DER 732 (30 pbw)	130°C@15h
Rcp6	GY282	Jeffamine D400/D2000	DER 732	80°C@2h 125°C@3h
Rcp7 ITER CS [2]	GY282	HY 918	DY 073	128°C@12h

[1] Reed, R.P., Low-Viscosity, Radiation-Resistant Resin System with Increased Toughness. 2004. 711: p. 209-216.
[2] Reed, R., D. Evans, and P. Fabian, Development of a new resin system for the US ITER central solenoid model coil, in Advances in Cryogenic Engineering Materials. 2000, Springer. p. 227-234.

Initial Screening test_Thermal shock test

Room T to LN_2 to Room T

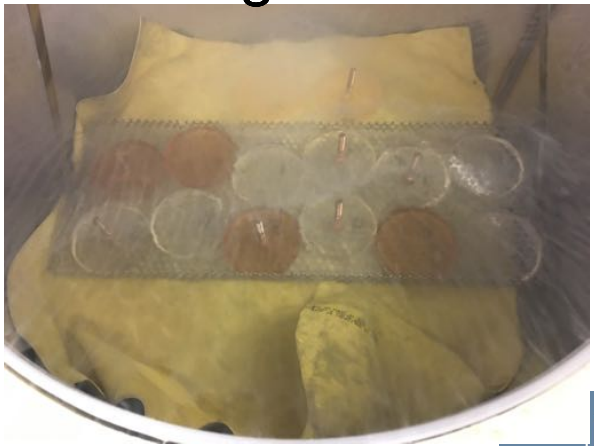
Size of the samples: Diameter 70 mm
Thickness 10 mm

Epoxy resin blocks
- contain an bronze screw
- without an bronze screw

14 samples were tested for 7 recipes.

Before the test

During the test



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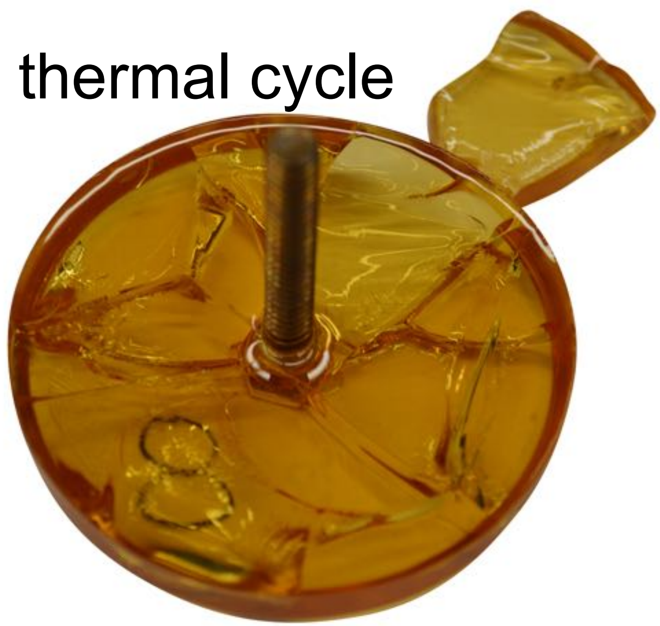
Thermal shock test   Rcp 4_ATLAS ECT

After one-time thermal cycle

After 10-times thermal cycle

After 20-times thermal cycle



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Thermal shock test ATLAS ECT

Rcp 5_Modified

After one-time thermal cycle

After 10-times thermal cycle

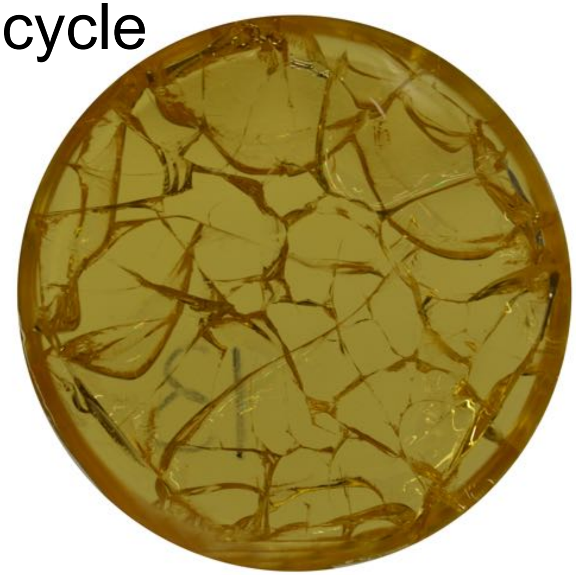
After 20-times thermal cycle

Thermal shock test Rcp 7_ITER CS cracks like its sibling CTD101k

After one-time thermal cycle

After 10-times thermal cycle

After 20-times thermal cycle

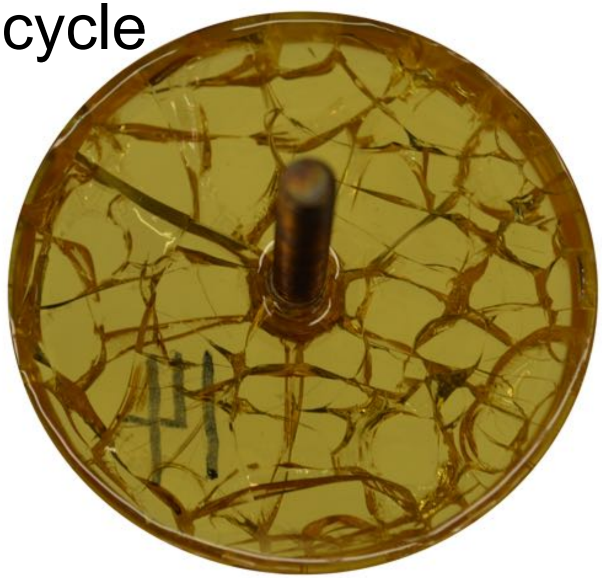


Thermal shock test Rcp 7_ITER CS

After one-time thermal cycle

After 10-times thermal cycle

After 20-times thermal cycle



Initial Screening test_Thermal shock test

After 20-times thermal cycle

Samples with screw

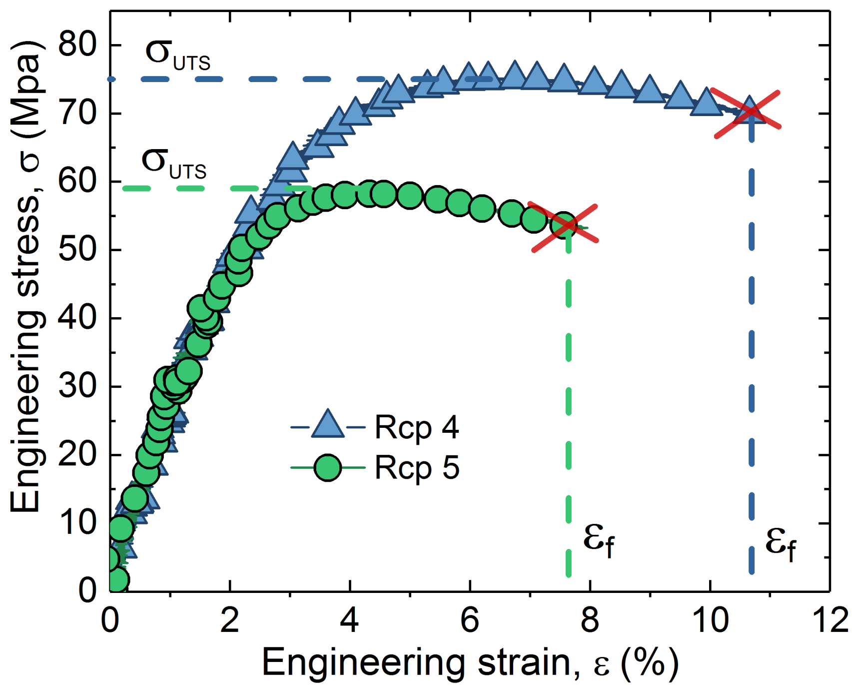
Recipe No.	Sample No.	Crack at first cycle	Cycles prior to failure
Rcp1	2	N	>20
Rcp2	4	N	>20
Rcp3	6	N	>20
Rcp4 ATLAS ECT [1]	8	N	10
Rcp5 ATLAS ECT [1]	10	Y	0
Rcp6	12	N	>20
Rcp7 ITER CS [2]	14	Y	0

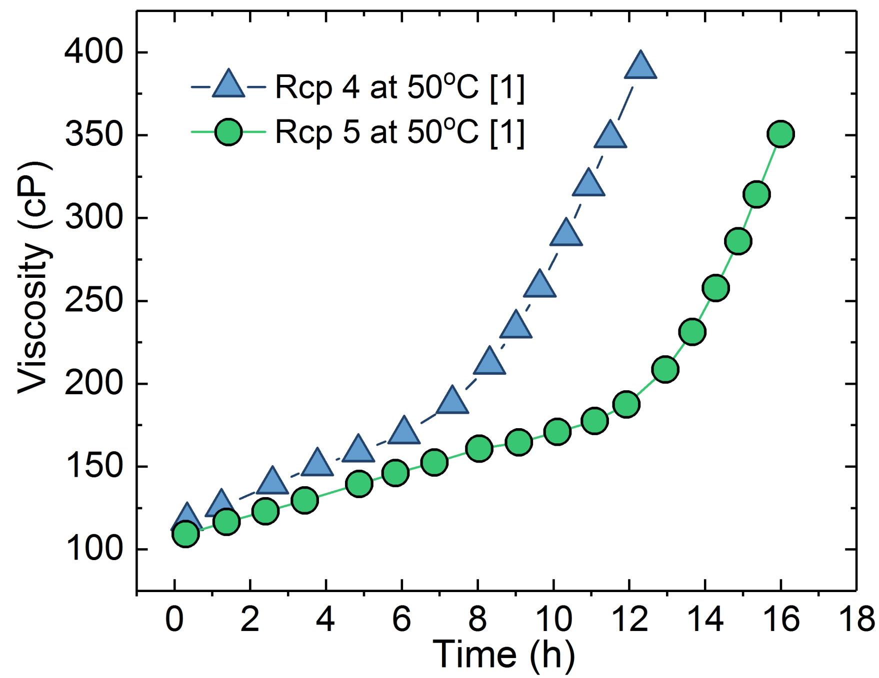
[1] Reed, R.P., Low-Viscosity, Radiation-Resistant Resin System with Increased Toughness. 2004. 711: p. 209-216.
[2] Reed, R., D. Evans, and P. Fabian, Development of a new resin system for the US ITER central solenoid model coil, in Advances in Cryogenic Engineering Materials. 2000, Springer. p. 227-234.

Tensile test at RT – ATLAS ECT and its modified version show some ductivity.

Recipe No.	Epoxy	Curing agent	Weight ratio (Epoxy : Curing agent : Modifier)
Rcp 4 ATLAS ECT [1]	GY282	HY5200	90 26 (HY5200) 10 (DER 732)
Rcp 5 ATLAS ECT [1]	GY282	HY5200	70 22 (HY5200) 30 (DER 732)

Higher stress and strain → Lower viscosity and shorter pot life

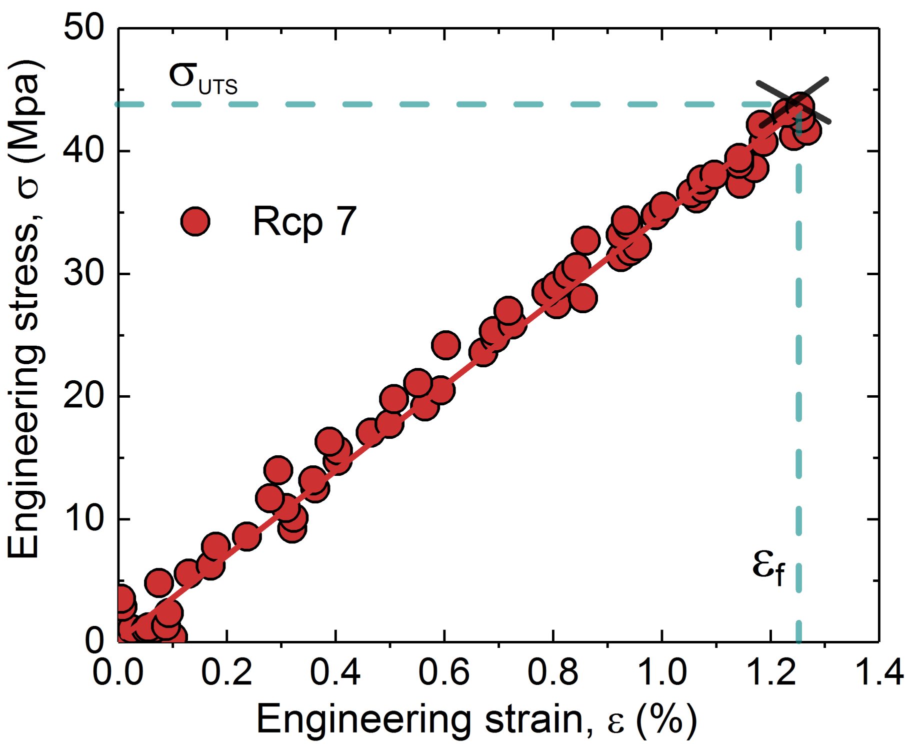


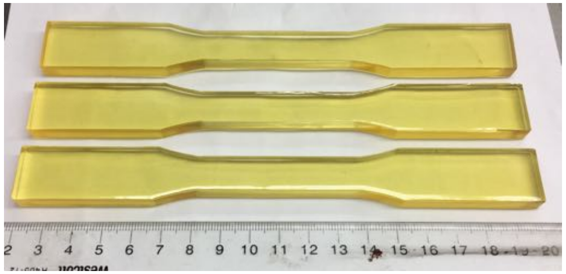


[1] Reed, R.P., Low-Viscosity, Radiation-Resistant Resin System with Increased Toughness. 2004. 711: p. 209-216.

Tensile test at RT – ITER CS epoxy, like its sibling CTD101k, is brittle

Sample No.	Epoxy	Curing agent	Weight ratio (Epoxy : Curing agent : Modifier)
Rcp 7 ITER CS [1]	GY282	HY 918	100 82(HY 918) 0.25 (DY073)





[1] Reed, R., D. Evans, and P. Fabian, Development of a new resin system for the US ITER central solenoid model coil, in Advances in Cryogenic Engineering Materials. 2000, Springer. p. 227-234.

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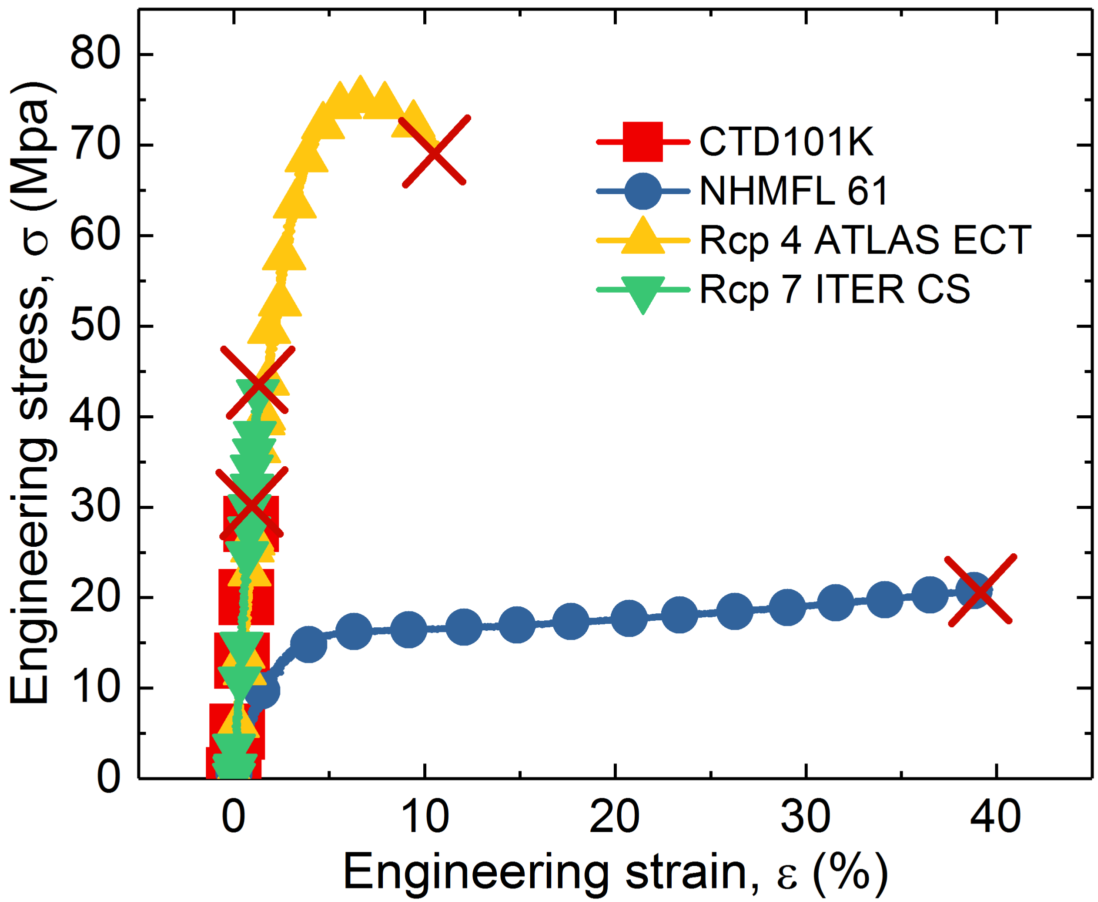
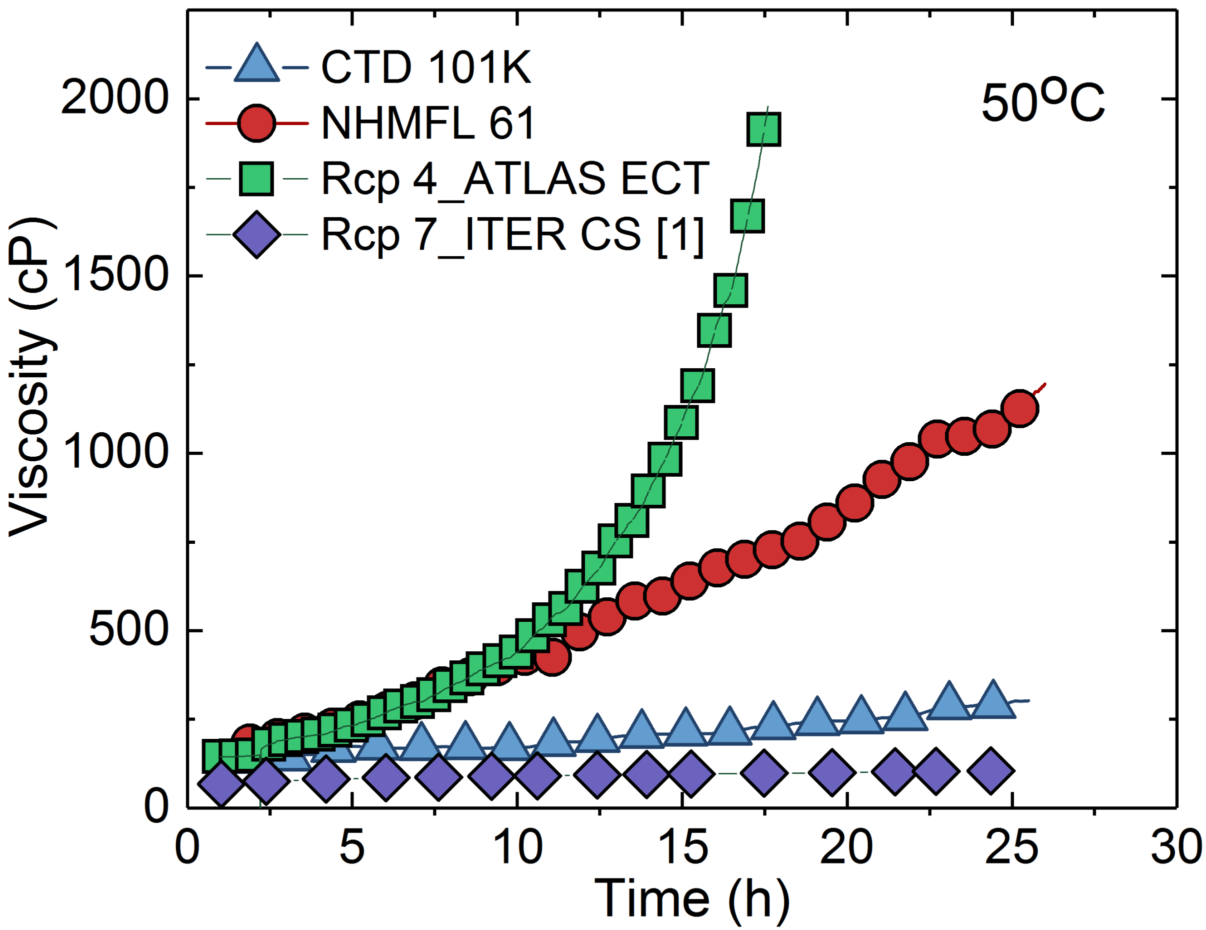
Tensile test comparison

CTD101 K, NHMFL mix-61, Rcp4_ATLAS ECT epoxy, and Rcp7_ITER CS



Viscosity and pot life comparison

CTD101 K, NHMFL mix-61, ATLAS ECT epoxy, and ITER CS epoxy at 50C



[1] Madhukar, Madhu S., and Nicolai N. Martovetsky. "DGEBF epoxy blends for use in the resin impregnation of extremely large composite parts." Journal of Composite Materials 49.30 (2015): 3741-3753.

Glass transition temperature comparison

Recipe No.	Epoxy	Curing agent	Modifier	Tg (^oC)
Rcp1	GY282	Jeffamine D230	N/A	44
Rcp2	GY282	Jeffamine D400	N/A	32
Rcp3	GY282	Jeffamine D400/D2000	N/A	N/A
Rcp4 ATLAS ECT [1]	GY282	HY5200	DER 732 (10 pbw)	123
Rcp5 ATLAS ECT [1]	GY282	HY5200	DER 732 (30 pbw)	68
Rcp6	GY282	Jeffamine D400/D2000	DER 732	N/A
Rcp7 ITER CS [2]	GY282	HY 918	DY 073	85
CTD 101K				105
NHMFL 61				65

Summary and recommendations

Virtually all Nb_3Sn accelerator magnets have been potted with CTD-101k or its siblings.

- **Most of them have long quench training.**

Test the NHMFL mix-61 and the Rcp 4_ATLAS ECT epoxy, two high toughness epoxy on both cosine-theta and CCT windings.

- **Reasonable pot life and viscosity at 50C and reasonable curing temperatures.**
- **High thermal shock resistance**
- **High toughness and elongation at break**
- **For cosine-theta magnets, they might benefit from pressure VPI.**

Still testing new recipes to add into our toolbox.